INTERMITTENT CONTACT IMAGING UNDER FORCE-FEEDBACK CONTROL

Field Of The Invention

The present invention relates generally to imaging and in particular to a method and apparatus for intermittent contact imaging.

Background Of The Invention

Atomic-force microscopy has become widely used to image surfaces of samples on a microscopic scale. Its popularity to a large extent is due to the fact that an atomic-force microscope (AFM) measures the force or force gradient between a sharp tip disposed on a cantilever and a sample surface at a picoNewton (pN) level as opposed to the tunneling current measured with a scanning tunneling microscope (STM). This of course allows the AFM to image insulating as well as conducting samples.

AFMs can be operated in either contact or intermittent contact modes. When operating in a contact mode, the deflection of a weak cantilever is kept constant while servoing the vertical extension of a piezoelectric scanner supporting the sample being imaged. The piezoelectric scanner is also rastered in an x-y plane to scan the surface of the sample. A map of the vertical extension of the piezoelectric scanner at various x,y coordinates of the sample surface, which is assumed to be proportional to a change in voltage on the piezoelectric scanner, reflects the topography of the sample surface. Unfortunately, problems exist in that soft samples are often damaged by the plowing action of the tip on the sample as the sample is rastered by the piezoelectric scanner in the x-y plane.

When operating in an intermittent contact mode, the base of a stiff cantilever is driven by a piezoelectric element which induces an oscillation at the free end of the cantilever. By driving the cantilever near its resonant frequency, an oscillation amplitude ranging from 20 to 100 nm at the free end of the cantilever can be achieved. This amplitude range is sufficient to inhibit the tip from sticking to the sample surface during each contact. To generate the image, the vertical extension of the piezoelectric scanner is servoed to maintain a constant drop in the oscillation amplitude. The piezoelectric scanner is also rastered in an x-y plane to scan the

surface of the sample. In order to achieve high sensitivity, a high quality factor (Q) is necessary. Tapping mode cantilevers typically have Q values ranging from 100 to 1000 in air.

To enhance the measurement of force displacement curves, a modified form of atomic-force microscopy, referred to as interfacial force microscopy, has been developed. Interfacial force microscopes (IFMs) replace the cantilever with a differential-capacitance displacement sensor. Feedback is used to servo the net electrostatic torque of the sensor such that it cancels the torque resulting from tip-sample forces. As a consequence, the tip support remains at its rest position throughout the force profile. This feature eliminates the snap-to-contact instability which plagues weak cantilevers in the attractive force regime and correlates the tip-sample deformation directly to the vertical extension of the piezoelectric scanner in nanoindentation studies.

Feedback attempts to inhibit the common plate of the displacement sensor from actually deflecting which leads to rapid restabilization of the displacement sensor after hard collisions with pronounced surface features. However, this places considerable demands on the rate at which force signals drift since it is often necessary to image a large field of view at a slow lateral scan. Generally, a contact force in the range of 200 nN corresponds to a force signal in the order of 10mV. As will be appreciated, the force signal has very little room to drift over the scan duration. A slow drift in the attractive force direction results in a slight increase in the contact force applied to the sample over the scan. Drifts in the repulsive force direction can pull the piezoelectric scanner completely out of feedback. Accordingly, improved imaging techniques are desired.

It is therefore an object of the present invention to provide a novel method and apparatus for intermittent imaging.

Summary Of The Invention

According to one aspect of the present invention there is provided an apparatus for intermittent contact imaging comprising:

a sensor to contact intermittently a sample to be imaged and generating displacement signals during oscillation thereof;

a scanner adjacent said sensor and supporting said sample to be imaged, said scanner being actuable to move said sample relative to said sensor to bring said sensor into intermittent contact with said sample; and

a controller in communication with said sensor and said scanner, said controller including a sensor feedback circuit receiving said displacement signals and an AC setpoint signal, said AC setpoint signal having a frequency generally equal to the frequency at the peak of the displacement versus frequency curve of said sensor, the output of said sensor feedback circuit being applied to said sensor to oscillate the same, said controller further providing output to said scanner in response to said displacement signals to control the separation distance between said sensor and said sample.

According to another aspect of the present invention there is provided an interfacial force microscope comprising:

a differential-capacitance displacement sensor having a tip mounted on an oscillating member, said sensor generating displacement signals during oscillation of said member;

a scanner adjacent said sensor and supporting a sample to be imaged, said scanner being actuable to move said sample relative to said sensor to bring said tip into intermittent contact with said sample and to move said sample relative to said sensor to raster said sensor over said sample; and

a controller in communication with said sensor and said scanner, said controller including a sensor feedback circuit receiving said displacement signals and an AC setpoint signal, said AC setpoint signal having a frequency generally equal to the frequency at the peak of the displacement versus frequency curve of said sensor, the output of said sensor feedback circuit being applied to said sensor to oscillate said member, said controller further providing output to said scanner in response to said displacement signals to control the separation distance between said sensor and said sample.

According to still yet another aspect of the present invention there is provided a method of imaging a sample surface comprising the steps of:

oscillating a sensor at a driven setpoint frequency to cause said sensor to intermittently contact a sample to be imaged;

generating displacement signals in response to oscillations of said sensor;

moving the sample relative to said sensor to maintain the separation distance between said sensor and sample; and

rastering said sensor over the sample surface, wherein said driven setpoint frequency is generally equal to the frequency at the peak of the frequency versus displacement curve of said sensor.

The present invention provides advantages in that soft samples can be imaged on a microscopic level using a highly damped sensor while reducing the shear forces applied to the sample as the sample is scanned.

Brief Description Of The Drawings

An embodiment of the present invention will now be described more fully with reference to the accompanying drawings in which:

Figure 1 is a schematic block diagram of an interfacial force microscope under force-feedback control configured to operate in an intermittent contact mode;

Figure 2 is another schematic block diagram of the interfacial force microscope of Figure 1 showing further detail;

Figure 3 is yet another schematic block diagram of the interfacial force microscope of Figure 1 showing further detail;

Figure 4 is an enlarged, exploded perspective view of a differential-capacitance displacement sensor forming part of the interfacial force microscope of Figure 1;

Figure 5 is a schematic block diagram of a force-feedback controller forming part of the interfacial force microscope of Figure 1;

Figure 6 is a block circuit diagram of a PID controller forming part of the force-feedback controller of Figure 5;

Figure 7 shows a 1kHz intermittent contact image of Kevlar fiber-epoxy taken using the interfacial force microscope of Figure 1;

Figure 8 shows another 1kHz intermittent contact image of Kevlar fiber-epoxy taken using the interfacial force microscope of Figure 1 highlighting a damaged area; and

Figure 9 shows graphs illustrating the magnitude and phase of the relationship between the ratio of V_{demod} and V_{aux} as a function of frequency when the differential-capacitance displacement sensor is operated in air;

Figure 10 shows graphs illustrating the magnitude and phase of the relationship between the ratio of V_{PID} and V_{aux} as a function of frequency when the differential-capacitance displacement sensor is operated in air;

Figure 11 shows graphs illustrating the magnitude and phase of the relationship between the open loop gain GOL as a function of frequency when the differential-capacitance displacement sensor is operated in air with the curves of Figure 8 superimposed thereon; and

Figure 12 shows intermittent contact images of the surfaces of Hexadecane (3.34cP) and Glycerol (1490cP).

Detailed Description Of The Preferred Embodiment

Referring now to Figure 1, an interfacial force microscope (IFM) under force-feedback control and configured to operate in an intermittent contact mode to generate microscopic images of a sample under observation in accordance with the present invention is shown and is generally indicated to by reference numeral 20. As can be seen, the IFM 20 includes a differential-capacitance displacement (DCD) sensor 22 to contact intermittently the sample to be imaged. A controller 24 is coupled to the DCD sensor 22 as well as to a piezoelectric scanner 26 positioned

adjacent the DCD sensor 22 and supporting the sample. The piezoelectric scanner 26 is responsive to the controller 24 to move the sample in a vertical direction to alter the sensor-sample separation. The piezoelectric scanner 26 is also actuable to move the sample laterally in an x-y plane to raster the DCD sensor 22 over the sample at a rate equal to about 2 to 3 μm/s. An imager 27 such as a NanoScope IIIa MultiMode manufactured by Digital Instruments receives output from the controller 24 and generates surface images of the sample. The controller 24 drives the DCD sensor 22 such that it operates in an intermittent contact mode while maintaining a high quality factor Q. The quality factor Q is of a value to achieve sufficient sensor output displacement signal contrast between out of contact and contact conditions of the DCD sensor 22 and the sample even though the DCD sensor is highly damped in air. Further details of the IFM 20 and in particular, the force-feedback control will now be described.

Figures 2 and 3 better illustrate the IFM 20. As can be seen, the controller 24 includes a force-feedback (FFB) controller 24a and an image feedback (IFB) controller 24b. FFB controller 24a is responsible for driving the DCD sensor 22 and includes a feedback circuit tuned to establish a well defined peak within the displacement-frequency spectrum of the DCD sensor 22 sufficient to achieve the desired high quality factor Q. The IFB controller 24b is responsible for servoing the vertical extension of the piezoelectric scanner 26 to control the sensor-sample separation at each x,y coordinate of the sample being intermittently contacted by the DCD sensor 22. The FFB controller 24b is connected to the DCD sensor 22 directly as well as through a preamplifier 28. The preamplifier 28 has a high input impedance to inhibit excessive loading on the DCD sensor 22.

An amplitude and phase detector 30, a function generator 32, an oscillator 34 and optionally a volt meter 36 are also connected to the FFB controller 24a. The IFB controller 24b is connected to the amplitude and phase detector 30 and provides output to the piezo-controller 26a of the piezoelectric scanner 26. The piezo-controller 26a in turn drives the piezoelectric tube 26b of the piezoelectric scanner in the z-direction to alter the sensor-sample separation.

A multi-channel analog to digital converter (ADC) 44 is connected to the IFB controller 24b, the FFB controller 24a and the amplitude and phase detector 30. The ADC provides the output to the imager 27. An optional oscilloscope 46 is connected to the FFB and IFB controllers 24a and 24b respectively.

Turning to Figures 2 to 4, the DCD sensor 22 is better illustrated. As can be seen, the DCD sensor 22 includes a stainless steel or Beryllium-Copper (BeCu) common plate 50. A common plate 52 having a pair of torsion bars 54 extending in opposite directions from its sides is defined by a cut 56 in the common plate 50. The common plate 52 is disposed above a pair of gold or chromium electrodes 58 mounted on a quartz substrate 60. A tip 62 is attached to the common plate 52 by way of a conductive adhesive to help to reduce the mass of the DCD sensor 22. The tip 62 is fashioned from a wire having a diameter equal to about 0.125mm by electrochemical etching and has a parabolic profile. The common plate 52 is connected to the input of the preamplifier 28.

The electrodes 58 are dc biased and are driven by RF driving signals output by the FFB controller 24a. The RF driving signals are 180 degrees out of phase and have a frequency well beyond the mechanical bandwidth of the DCD sensor 22 (i.e. 1MHz) to establish an RF capacitance bridge defined by the electrodes 58 and common plate 52 that is sensitive to changes in capacitance at an aF level. The electrodes 58 are also driven by an AC setpoint signal generally in the range of from about 1 kHz to 1.5 kHz which causes the common plate 52 to oscillate as will be described. The frequency of the AC setpoint signal is a function of the mechanical properties of the DCD sensor material, its dimensions etc.

When the tip 62 encounters the sample supported by the piezoelectric scanner 26, a force is applied to the tip 62 resulting in torque being applied to the common plate 52. The applied torque causes the common plate 52 to rotate. Rotation of the common plate 52 changes the capacitance between the electrodes 58 and the common plate 52 and is detected by the RF capacitance bridge. When the common plate 52 rotates by a small angle $\theta \approx \delta/L$ where δ is the change in the average gap between the common plate 52 and one of the electrodes 58 and L is the distance

between the mid-point of the torsion bar axis and the tip 62, a displacement signal appears on the common plate 52. The displacement signal has an amplitude equal to $2\Delta CV_{ac}/C_{Total}$, where ΔC is the change in capacitance, V_{ac} is the amplitude of the RF driving signals and C_{Total} is equal to two times the capacitance between the common plate 52 and one electrode 58 plus any stray capacitance in parallel with the DCD sensor 22. The displacement signal has a frequency equal to the frequency of the RF driving signals and a phase dependent on the direction of rotation of the common plate 52. The displacement signal on the common plate 52 is picked up by the preamplifier 28, amplified and conveyed to the FFB controller 24a.

Figure 5 better illustrates the FFB controller 24a. As is shown, FFB controller 24a includes a demodulator 70 receiving the displacement signal output of the preamplifier 28 and the RF signal output of the function generator 32. The demodulator 70 demodulates and low pass filters the displacement signal output of the preamplifier 28 to generate demodulator amplitude signal output V_{demod}. The demodulator signal output V_{demod} is applied to the amplitude and phase detector 30 and to a PID controller 72. The PID controller 72 also receives the AC setpoint signal output of the oscillator 34 and generates V_{PID} and $-V_{PID}$ feedback signals. The $-V_{PID}$ feedback signal is conveyed to one of the channels of ADC 44 and to the oscilloscope 46 while the V_{PID} feedback signal is conveyed to an RF PID controller 74. The RF PID controller 74 also receives a DC voltage and the RF signal output of the function generator 32 from the demodulator 70 and supplies the RF driving signals to each of the electrodes 58.

The PID controller 72 is better illustrated in Figure 6 and as can be seen, it includes a summing amplifier 80 having unity gain. The summing amplifier receives the demodulator signal output V_{demod} as well as the AC setpoint signal V_{aux} from oscillator 34. The sum output of the summing amplifier 80 is therefore $-(V_{aux} + V_{demod})$ and represents the error signal for the DCD sensor feedback loop. The sum output of the amplifier 80 is applied to a proportional-integral-derivative (PID) control block 82. The PID control block 82 has a good low frequency response and provides output proportional to the combination of its input, the time integral of its input and

the time rate of change of its input. The output of the PID control block 82 is conveyed to a pair of summing amplifiers 84 and 86 functioning as high-pass filters, one amplifier 84 of which generates the V_{PID} feedback signal and the other amplifier 86 of which generates the $-V_{PID}$ feedback signal. Since the summing amplifier 80 has unity gain, the gain G_{PID} from the input of the PID control block 82 to the output of the summing amplifier 84 can be expressed as:

$$G_{PID} = -(G_P + G_I + G_D)$$

where:

G_P is the proportional gain and is equal to -1;

G_I is the integrator gain and is equal to j/wT_I;

G_D is the derivative gain and is equal to $G_D = -\Gamma[j\omega T_D/(1+j\omega T_D)]$ over the frequency range of interest;

T_I is the time constant of the integrator;

T_D is the time constant of the differentiator; and

Γ represents the gain at the end of the high-pass filter circuit.

The gain term G_{PID} allows the frequency response of the DCD sensor feedback loop to be tailored.

If the frequency of the AC setpoint signal V_{aux} is near dc, the setpoint signal V_{aux} acts as the driven setpoint of the DCD sensor causing the DCD sensor 22 to oscillate physically such that the resulting feedback signal V_{demod} output by demodulator 70 cancels the setpoint signal V_{aux}. In this case, the resulting error signal $-(V_{aux} + V_{demod})$ is basically equal to zero. When the AC setpoint signal V_{aux} is moved to higher frequencies, the feedback system is unable to offset the AC setpoint signal V_{aux} resulting in error signals which can be quite large.

Prior to imaging, the electronic gains of the integrator and differentiator of the PID controller 72 are adjusted such that the sensor displacement signal is nearly in-phase with the AC setpoint signal V_{aux} at the frequency where the open loop gain falls to one (1). In other words, the feedback system has very little phase margin before it becomes unstable. However, the feedback system exhibits a much higher quality factor Q then if operated without feedback. The PID controller

gain adjustments are chosen to obtain a quality factor Q high enough to be sensitive to perturbations caused by the tip striking the sample but not so large that noise becomes an issue. Noise becomes an issue when the quality factor Q is increased to a point where the phase margin becomes so small that the feedback system edges too close to the brink of instability.

Once tuning of the PID controller 72 has been completed, the displacement vs. frequency plot of the DCD sensor 22 is examined to find the frequency of maximum displacement. The frequency of the setpoint signal V_{aux} is then set to the frequency of maximum displacement so that the DCD sensor 22 oscillates at this frequency. At this time, the piezoelectric scanner 26 is actuated to bring the sample towards the DCD sensor 22 so that the tip 62 intermittently contacts the sample as the common plate 52 oscillates. When the tip 62 contacts the sample, the amplitude of the DCD displacement signal decreases.

The amplitude and phase detector 30 applies the amplitude signal V_{demod} to the IFB controller 24b which in turn outputs a magnitude signal to the piezo-controller 26a controlling the piezoelectric tube 26b. The imaging set point is set about 3% lower than the initial output of the amplitude and phase detector 30. The piezoelectric scanner 26 in turn moves the sample towards the DCD sensor 22 to control the separation between the tip 62 and the sample such that the displacement signal of the DCD sensor 22 is constant but lower than the in-air case. During this process, the piezoelectric scanner 26 is rastered in an x-y plane to image the surface of the sample under observation.

The error signals of the piezoelectric scanner feedback loop are applied to the ADC 44 which also receives phase input from the amplitude and phase detector 30 and the V_{PID} feedback signal from the FFB controller 24a. The digital output of the ADC 44 is conveyed to the imager 27 to allow images to be formed. The error signals of the piezoelectric scanner feedback loop provide edge-contrast information of the sample surface topography while the phase of the displacement signal provides information related to energy dissipation during tip and sample contact.

By keeping the mass of the DCD sensor 22 low and establishing a well defined peak in V_{demod}, a high quality factor is maintained. This allows the DCD sensor to be operated in an intermittent contact mode while ensuring sufficient contrast between out of contact and contact displacement signals generated by the DCD sensor. As a result, high quality images of the sample under observation can be generated at a microscopic level.

For a test sample, a fiber composite comprised of Kevlar 49 fibers imbedded in an epoxy matrix (the fiber volume fraction is reported to be 50%) was imaged using the IFM 20. Prior to imaging, the sample was polished. Figure 7 shows a 1 kHz intermittent contact IFM image (all images: 180x180 points, plane subtraction, no filtering) of the fiber composite sample. The ~ 50 nm deep polishing grooves are clearly evident, in spite of a maximum height difference of ~ 740 nm. The piece of debris at the left edge remained undisturbed by the imaging procedure. The total imaging time was 11 min, corresponding to 20 contact cycles per point.

Figure 8 shows a 1 kHz intermittent contact IFM image of a badly damaged area on the Kevlar sample. The massive surface upheaval results in a maximum height difference approaching 3μm, which deaccentuates the shallow polishing grooves in the unblemished fiber regions. In spite of the upheaval, the intermittent contact mode technique had little difficulty in tracking the surface topography, although optimum imaging required doubling the number of contact cycles per point.

To determine the peak contact force during imaging, the separation between the tip 62 and a single Kevlar 49 fiber was narrowed until the first evidence of intermittent contact was observed, and then the piezoelectric tube 26b was advanced until the set point was reached. After dividing the amplitude and phase detector output by the appropriate gain terms, it was estimated that the oscillation amplitude of the common plate 52 was reduced from 18.97 nm in air to 18.36 nm during intermittent contact (amplitudes being expressed as peak-to-peak values). The distance that the piezoelectric tube 26b advanced to reach the set point was 9.9 Å,

which is to be compared to the 6.1 Å reduction in the common plate oscillation amplitude.

Figure 12 shows intermittent contact images of the surfaces of Hexadecane (3.34cP) and Glycerol (1490cP). As will be appreciated, the DCD sensor 22 can be controlled under force-feedback to image soft as well as hard samples.

In the intermittent contact mode, the motion of the common plate 52 decays only during the contact portion of the cycle. Therefore, the difference between the advancement of the piezoelectric tube 26b and the reduction in common plate oscillation amplitude is a reasonable estimate of the maximum tip-sample deformation, which is about 3.8 Å.

To estimate the peak contact force, the Hertz equation for elastic contact which is appropriate for axis symmetric parabolic bodies is used:

$$F = \frac{4}{3} E^* \sqrt{R} * D^{3/2}$$

where:

$R = (1/R_t + 1/R_s)^{-1}$

$E^* = [(1-v_t^2)/E_t + (1-v_s^2)/E_s]^{-1}$ and is the reduced modulus;

D is the combined deformation of the tip and sample;

v_t, v_s refer to Poisson's ratio;

E_t, E_s indicate Young's modulus;

R_t, R_s represent parabolic radii of curvature; and

t, s denote tip and sample.

To test the sensor feedback loop, the ratio V_{demod}/V_{aux}, and the ratio V_{PID}/V_{aux}, as a function of frequency were measured with the DCD sensor 22 operating in air. It is easy to show that theoretically $V_{demod}/V_{aux}=G_{OL}/(1-G_{OL})$ and $V_{PID}/V_{aux}=G_{PID}/(1-G_{OL})$, where $G_{OL}=-G_{PID}G_{force}G_{mech}G_{bridge}G_{preamp}G_{demod}$ is the open loop gain, the minus sign being a result of the summing amplifier 80. Figures 9 and 10 show how these two ratios vary with frequency when the DCD sensor 22 operates under the following set of conditions:

$T_I=4.3\text{x}10^{-5}$ s; $T_D=3.6\text{x}10^{-5}$ s; $\Gamma=2.4$; and $G_{demod}=4.1$

G_{demod} is referenced to the peak-to-peak amplitude of the preamplifier output. The solid line passing through the experimental points is the theoretical result. The level of agreement between theory and experiment ranges from good to excellent.

Of note in the magnitude plots is the presence of the well-defined peak occurring around 660 Hz signifying that the sensor feedback loop behaves much like a second-order low-pass filter. The origin of this behavior is rooted in Barkhausen's criterion for feedback stability. In other words, the term $1/(1-G_{OL})$ tends to infinity if the magnitude of G_{OL} approaches unity and the corresponding phase approaches zero.

Figure 11 shows the calculated frequency dependence of G_{OL} with the magnitude plot for V_{demod}/V_{aux} superimposed. As can be seen, the magnitude of G_{OL} is roughly 0 dB (or 1) and the corresponding phase is about 0.08Π at the peak frequency for demodulator output signal V_{demod}. The phase margin is small enough to obtain a strong resonance response, but large enough to prevent the sensor feedback loop from going into self-oscillation. It is important to note that a peak in the demodulator output signal V_{demod} does not mean mechanical resonance. In the example shown, the peak frequency for the demodulator output signal V_{demod} is in fact 60 Hz lower than $\omega_0/2\Pi$, the mechanical resonant frequency of the DCD sensor 22. Nevertheless, a maximum in the demodulator output signal V_{demod} does mean a maximum in the displacement amplitude, but this is not achieved in the usual way. Looking at the magnitude plot for V_{PID}/V_{aux}, it can be seen that the applied force varies over the frequency range, and reaches a maximum in the vicinity of the peak frequency for the demodulator output signal V_{demod}.

A comparison between the phase plots shows that the phase of the displacement (or V_{demod}) lags the phase of the force (or V_{PID}) by an angle reasonably close to $\Pi/2$. This, along with the fact that a 60 Hz difference in frequency is not very large, suggests that ω_0 does play an important role in obtaining a strong peak, which can be understood in the following way. In order to obtain a strong peak and still maintain feedback stability, one must make G_I the dominant electronic gain term because it is the only electronic gain that rolls off its response with increasing frequency, which means that the phase lag due to the electronics is not far removed

from $\Pi/2$ over the frequency range of interest. The mechanical phase lag eventually reaches $\Pi/2$ at ω_0, which yields an overall phase lag in the neighborhood of the Π phase lag required to bring the phase of G_{OL} to zero.

As will be appreciated, the present invention provides advantages in that samples can be imaged on a microscopic level without damaging the samples. This makes the present imaging technique particularly suited to imaging soft samples including emulsions and liquids. Images can be taken for several hours without removing the varying dc offset in the force signal which is required during contact mode imaging to ensure minimal contact force between the tip and the sample.

Although the present invention has been described with specific reference to interfacial force microscopy and use of a differential-capacitance displacement sensor, those of skill in the art will appreciate that the feedback control used in the preset imaging technique can be used with other heavily-damped displacement sensors. It will also be appreciated by those of skill in the art, that variations and modifications may be made to the present invention without departing from the spirit and scope thereof as defined by the appended claims.